

TEXTILE FABRIC WITH PARTICLE ATTRACTING FINISH

This invention relates to textile fabrics having a particle attracting finish applied thereto. The fabrics are referred to as wipers. The wipers find utility in cleaning surfaces, whenever it is desirable to minimize particulate contamination.

Wipers are utilized for a number of different cleaning applications, such as in
5 cleanrooms, automotive painting rooms and other controlled environments. Each different application emphasizes certain standards that these types of wipers should attain. For example, wipers utilized in cleanrooms must meet stringent performance standards. These standards are related to sorbency and contamination, including maximum allowable particulate, unspecified extractable matter and individual ionic
10 contaminants. The standards for particulate contaminant release are especially rigorous and various methods have been devised to meet them.

Wipers may be made from knitted, woven or non-woven textile fabrics. The fabric is cut into wipers, typically 9 inch by 9 inch squares. The wipers may remain unlaundered or may be washed in a cleanroom laundry, employing special surfactants and
15 highly-filtered and purified water, to reduce the contamination present on the fabric. After washing, the wipers may be packaged dry in air-tight plastic bags, or pre-saturated with a suitable solvent before being packaged, and are ready for use.

Prior art developments in the field of wipers include reduction in particulate contaminants through the use of a specific yarn, such as "nylon bright" as disclosed in Paley et al., US 5,271,995, and reduction in loose fiber contamination by using a hot air jet to cut the fabric into pieces while simultaneously sealing the edges as disclosed in Reynolds US 5,069,735. Finishes to improve the sorbency of wipers made of hydrophillic fibers, such as polyester, have also been employed. Wiping cloths having a textile substrate and a porous polymer coating made from the "sulphonation products of cross-linked polymers containing sulphonated aromatic residues" are disclosed in GB 2 142 225 A.

Tack cloths are textile fabrics of relatively loose weave which have been chemically treated to give them a sticky or tacky character. They are used to remove dust from surfaces prior to applying a coating and for dusting in the home. Hansen, US 3,208,093, discloses a tack cloth having a plasticizer/vinyl polymer composition applied thereto in the amount of 10 to 16 parts polymer to about 11 parts of substrate. Bennet, US 3,658,578 discloses a fabric substrate impregnated with an amorphous polypropylene compound characterized by a m.w. of less than 10,000, to achieve a tacky finish.

Summary of the Invention

The objects of the present invention are to provide: a textile wiper with a particle attracting finish; a textile wiper suitable for use in cleanrooms and other controlled environments where the wiper itself must be low in contaminants; a particle attracting

wiper which will function in a wide range of applications - both dry and in conjunction with solvents; and a finish which is durable, yet exhibits little or no tack. In one embodiment, the wiper is laundered under conditions to leave a surfactant residue, which has been found to improve absorbency.

5 Accordingly, a textile fabric having a particle attracting, polymer finish is provided for use as a wiper. The wiper may be used dry or saturated with a desirable solvent. In one embodiment, the wipe is pre-saturated with a cleaning solvent and packaged in a resealable container.

10 The particle attracting potential of the present wiper has been characterized by a "particle attraction coefficient" measured by the following test:

 A 9" x 9" wiper is pre-wetted with water and placed in a beaker containing 400 ml of water and 40 mg (0.01% by weight) of carbon black having an average particle size of 3 microns, as measured by a Microtrac UPA-150 analyzer, and obtained from Cabot Corporation (USA), identified as carbon black M-1300. The wiper is stirred in the beaker
15 using a magnetic stir bar for 30 seconds and removed. Excess water in the wiper is squeezed out, draining the water back into the beaker. The water in the beaker is then filtered through a 1.0 micron pore size glass fiber filter. The amount of carbon black particulate which was left in the beaker is calculated, and the amount of carbon black particulate absorbed by the wiper can be determined. The test is repeated for the
20 identical fabric, without the particle attracting finish having been applied. The particle

attraction coefficient (%) is calculated using the following formula:

$$\frac{\text{wt. of particulate (fabric + finish)} - \text{wt. of particulate (fabric only)}}{\text{wt. of particulate (fabric only)}} \times 100$$

For example, the wiper having the finish of the present invention absorbed 33 mg
 5 of carbon black, while the fabric without the finish absorbed only 13 mg. The particle
 attraction coefficient is 154%.

The particle attracting polymer may be selected from compounds having pendent
 groups which (i) exhibit hydrogen bonding, such as hydroxy, hydroxyalkyl and carboxy
 groups; (ii) have acid-base reactive groups, such as $-\text{COOH}$, $-\text{NH}_2$, $-\text{SO}_3$, and $-\text{NO}_2$. In
 10 one embodiment of the invention, the particle attracting polymer is water-soluble and
 selected from:

(i) polysaccharides having a plurality of pendent groups selected from hydroxy,
 hydroxyalkyl and carboxy groups; and

(ii) polymers formed by vinyl polymerization, having a plurality of pendent groups
 15 selected from hydroxy, hydroxyalkyl, carboxy, amino and alkylamino groups;

wherein the article has a particle attraction coefficient of 50% or greater for carbon
 black, and a particle count of particles greater than 0.5 microns of 75 million particles per
 square meter or less as measured by Biaxial Shake Test IEST-RP-CP-CC004.2.

Detailed Description of the Invention

Without limiting the scope of the invention, the preferred embodiments and features are hereinafter set forth. Unless otherwise indicated, the following conditions apply: all parts and percentages are by weight; conditions are ambient, i.e. one atmosphere of pressure and 25° C; the term "average" means number-average; aliphatic hydrocarbons, including alkyl and alkylene radicals, comprise from 1 to 4 carbon atoms. Unless otherwise indicated, the particle attraction coefficient is measured using particles having an average size of approximately 3 microns.

All of the United States patents cited in the Specification are incorporated by reference.

The wipers of the present invention may be constructed from woven, knitted or non-woven fabric. Non-woven fabrics and their methods of manufacture are well known in the art. For example, non-woven fabrics may be wet laid, dry laid, spun bond, needle punched; with or without binders to stabilize them.

The fabric may be made from staple or continuous filament fibers, or yarns made from such fibers. Yarns having a wide variety of denier and filament count may be employed. By way of example, yarns having a denier to filament ratio of from 0.1 to 10, a denier of 15 to 250 with filament counts ranging from 10 to 250 may be employed. For certain cleaning applications, it is desirable to increase the abrasiveness of the fabric, and the fabric may incorporate filaments ranging in denier from 10 to 50.

The fiber may be selected from synthetic and natural fibers and blends thereof. For example, the fiber may be polyester, polyamide, polyolefin e.g. polyethylene, polypropylene and ethylene/propylene copolymer, acrylic, polyurethane, cellulosic, e.g. cotton, rayon and acetate, silk or wool, and blends thereof. Preferably, the fiber is polyester, polyamide or polyolefin. Most preferably, the wiper is woven or knitted from continuous filament, polyester yarn, for example, textured polyester yarn.

A wide range of fabric weights may be employed in the present invention. Typically, the fabrics used for cleanroom wipers have a weight of 1 to 16 ounces per square yard, preferably 2 to 9 ounces per square yard.

The fabric may be washed or scoured to remove spinning oils, dirt and other contamination prior to application of the particle attracting polymer. It is usually advantageous to heat set the fabric, either before or after application of the polymer, to provide dimensional stability. The fabric is preferably heat set at a temperature above what the yarns have previously experienced, after the initial spinning of the fiber. For example, polyester yarn may be heat set at a temperature of from 180° to 300° F. Preferably, the fabric lies flat when it is heat set.

Heat setting may advantageously be performed in a tenter frame oven, in which the fabric is held flat during heating and while it begins to cool. The temperature of the oven may be higher than the temperature actually experienced by the yarn, which will be a

function of the oven or dryer temperature profile, length and speed of the fabric through the oven.

The particle attracting polymer finish or coating may be applied by any suitable method used to apply a coating in the form of a solution, dispersion, emulsion or a particulate to a fabric substrate. By way of example, the polymer may be applied by padding, wash wheel, spraying, nip roll, knife blade or in a jet dyeing apparatus. Preferably, the polymer is in the form of an aqueous solution.

The coat weight (solids) may be 0.01 wt.% or greater based on the weight of the fabric. Typically the coat weight is not greater than 6 wt.%. Preferably, the coat weight is from 0.02 to 3 wt.%, most preferably from 0.05 to 1 wt.%, based on the weight of the fabric. The particle attracting polymer is cured by a method appropriate to the polymer and the form in which it is applied (liquid or solid), and any residual liquor is evaporated. For example, if the polymer is applied in the form of a solution, dispersion or emulsion, the coated fabric may be dried and cured in a tenter oven.

The particle attracting polymer may be selected from polymers which exhibit an affinity for one or more of the following types of particulate matter: carbon black, aluminum, aluminum oxide, copper, copper oxide, ferrite, graphite, iron, iron oxide, manganese, manganese oxide, silicon, silicon dioxide, titanium, titanium dioxide, tungsten, tungsten dioxide, zinc and zinc oxide, preferably an affinity for one or more of the following types of particulate matter: carbon black, copper, copper oxide, silicon,

silicon dioxide, tungsten and tungsten dioxide. The particle sizes of particular concern are those in the range of 0.5 to 20 microns.

The efficacy of a particular wiper in attracting and removing particulate contaminants, from a surface or liquor, is referred to herein as a "particle attraction coefficient." The test has been previously described. Briefly, the particle attraction coefficient is the ratio of the increased particle attracting characteristics of a polymer coated wiper, relative to an uncoated wiper, for a given particle type. Preferably, the coated wiper of the present invention has a particle attraction coefficient for 1-5 micron size particles of 50% or greater, most preferably, 100% or greater.

Examples of particle attracting polymers include the following:

- (i) cellulose ethers, e.g. hydroxyalkylcelluloses, such as hydroxyethylcellulose and hydroxypropylcellulose; hydroxyalkylalkylcelluloses, such as hydroxyethylethylcellulose, hydroxybutylmethylcellulose, hydroxypropylmethylcellulose and hydroxyethylmethylcellulose; alkylcelluloses, such as methylcellulose and ethylcellulose; carboxyalkylcelluloses, such as sodium carboxymethylcellulose;
- (ii) inorganic cellulose esters, e.g. cellulose nitrate;
- (iii) chitosan; and

- (iv) guar gums and hydroxy, hydroxyalkyl and carboxy substituted derivatives thereof; e.g. carboxymethylguar gum, carboxymethyl(hydroxypropyl)guar gum, hydroxyethylguar gum and hydroxypropylguar gum.
- (v) starch and hydroxy, hydroxyalkyl and carboxy substituted derivatives thereof; e.g. hydroxyethylstarch and hydroxypropylstarch;
- (vi) poly(vinyl alcohol) and vinyl alcohol copolymers, e.g. ethylene/vinyl alcohol copolymers and vinyl alcohol/methacrylate or methylmethacrylate copolymers;
- (vii) poly(vinyl pyrrolidone);
- (viii) poly(hydroxyalkyl acrylate) and poly(hydroxyalkyl methacrylate), e.g. poly(hydroxypropyl methacrylate), poly(hydroxypropyl acrylate); and
- (ix) poly(alkyl acrylamide) and poly(alkyl acrylamide) copolymers, e.g. poly(isopropyl acrylamide) and isopropyl acrylamide/acrylic acid copolymer.

The class of suitable particle attracting polymers includes compounds having a broad range of molecular weights and solubilities in water. By way of example, polymers having an average molecular weight of from 1,000 to 2,000,000, preferably 25,000 to 1,000,000, most preferably 50,000 to 500,000 may be employed. Preferably, the polymer is water soluble, defined as a solubility of one part per 100 parts of water or greater, before the polymer is applied to the fabric and cured. Nevertheless, it is also desirable that the polymer, once applied to the fabric and cured, has sufficient durability to withstand laundering, such as in a cleanroom laundry. The particle attracting polymer

may also be applied to the fabric with a binder, preferably in minor amounts, such as melamine formaldehyde resin binder in conjunction with chitosan.

It is believed that in most cases, the polymer forms a film on the fibers of the textile fabric. The objects of the invention may be achieved, however, whether the
5 polymer forms a continuous film or is discontinuous.

In addition to the particle attracting polymer, other finishes may be applied to the textile fabric. For example, the textile fabric may be treated with a "soil release" finish to improve its wettability and washability, such as may be found in the following U.S.
10 patents: Marco, US 4,131,550; Hauser, US 4,164,392; Marco, US 4,168,954; Marco, US 4,170,557; Marco, US 4,235,735; Kimbrell, US 4,329,389; Schuette, US 5,725,951.

Examples of other compounds and compositions which may be applied to the textile fabric or used in conjunction with the particle attracting polymer include: plasticizers, antistatic agents, defoamers, anti-microbial or anti-fungal agents, lubricants, knitting oils and abrasives.

15 After the coated fabric is cured, the fabric is cut into nominal sizes for use as a cleanroom wiper, which are typically squares ranging from 4 inches by 4 inches to 24 inches by 24 inches. Any geometry may be employed, however. The fabric is preferably, though not necessarily, cut using a technique which fuses the end of the yarn, thereby preventing unraveling and particle generation. Examples of suitable techniques
20 may be found in Reynolds, US 5,069,735, and the references cited therein.

The wipers are typically packaged in a sealed container to keep them free from contamination.

For cleanroom applications, it is desirable to wash the fabric or wipers in a cleanroom laundry, which may be characterized as a laundry facility to remove and minimize contamination of the wipers, prior to packaging. The cleanroom laundry may employ special filters, surfactants, sequestrants, purified water, etc. to remove oils, reduce particle count and extract undesirable ion contaminants. The laundering process should not be overly aggressive, as the particle attracting finish may be removed.

Depending on the equipment employed, it may be necessary to adjust the agitation, volume and duration of rinsing and the speed and duration of extraction. Examples of suitable equipment and description of cleanroom laundries may be found in Austin, Dr. Philip R., "Encyclopedia of Cleanrooms, Bio-Cleanrooms and Aseptic Areas", Contamination Control Seminars, Michigan (1995).

In one embodiment, a surfactant residue is left on the wiper from the laundering process, and has been found to improve absorbency. The surfactant or surface-active agent may be selected from cationic, anionic, nonionic and ampholytic surfactants. A comprehensive description of surfactants finding utility herein may be found in the Kirk-Othmer Encyclopedia of Chemical Technology, 4th edition, pp. 478-541 (1997). Preferably, the surfactant is selected from anionic and nonionic surfactants.

A surfactant residue in the range of 0.00001 wt.% (0.1 ppm) to 0.5 wt.%, preferably 0.00005 wt.% (0.5 ppm) to 0.1 wt.%, has been found to significantly improve the absorbance of the wiper, especially with regard to polar solvents, such as alcohols and water. High levels of residue on the wiper may act as a source of contamination and are to be avoided.

For many applications, such as wipers for use in automotive paint rooms, it is not necessary to wash the wipers in a cleanroom laundry prior to use. Accordingly, the wipers may be packaged in a sealed container, without having been laundered subsequent to having been coated.

The wipers may be presaturated with a desired solvent and sold in sealed dispensers, as is well known in the art. The terms saturated and presaturated are used in their broad sense, i.e. wet with solvent. Suitable solvents include water, organic solvents such as naphtha, and aqueous solutions of water miscible organic solvents, in particular solutions of alcohols, such as C₁-C₈ alcohols. Of particular interest are wipers presaturated with a solution of isopropanol and water, for example, aqueous solutions containing 1 to 99 wt.% isopropanol. The solvent composition may also contain a surfactant and/or other additives selected for their cleaning characteristics. By way of example, additional solvents and packages for pre-saturated wipers may be found in the following references: US 3,994,751; US 4,627,936; US 4,639, 327; US 4,998,984; US

5,145,091; US 5,344,007 and JP 6[1994]-48475. Alternatively, the wipers may be sealed in air tight packages while dry.

Standards for Cleanroom Wipers

Among the standards which may be imposed on cleanroom wipers include performance criteria related to sorbency and contaminates. One standard for evaluating cleanroom wipers is the Institute of Environmental Sciences & Technology (IEST), Contamination Control Division Recommended Practice 004.2, which may be cited as IEST-RP-CC004.2, "Evaluating Wiping Materials Used in Cleanrooms and Other Controlled Environments".

Section 7 of Recommended Practice 004.2 sets forth some of the tests utilized for determining the capacity and rate sorption of cleanroom wipers. The capacity tests is performed by saturating a known area of wiper with a selected liquid and then calculating the volume sorbed per unit mass and per unit area of wiper (IEST-RP-CC004.2 § 7.1). The sorbency per unit mass is referred to as the "intrinsic sorbency" and is the volume of liquid in milliliters sorbed per unit of mass of wiper in grams. The "extrinsic sorbency" is the volume of liquid in milliliters sorbed per unit area of wiper in square meters.

The rate of sorption of a cleanroom wiper is measured by allowing a drop of water to fall from a fixed height onto the surface of a wiper. The time required for the disappearance of specular reflection from the drop is measured and recorded as the sorption rate (IEST-RP-CC004.2 § 7.2).

The primary test for contamination associated with cleanroom wipers are those measuring particles, unspecified extractable matter, and individual ionic constituents.

The number of particles released during wetting and mechanical stress can be measured in the Biaxial Shake Test (IEST-RP-CC004.2 § 5.2). Briefly, the wipers are placed in a jar of water and shaken. Aliquots are removed from the shaker and the number of particles is counted, typically those in the size range of 0.1 microns and larger are specified. The number of particles greater than a given particle size are reported in millions per square meter of fabric.

The amount of extractable contamination associated with a cleanroom wiper is determined by extracting the wiper with a solvent, such as water, isopropyl alcohol or acetone, evaporating the solvent and weighing the non-volatile residue (IEST-RP-CC004.2 § 6.1). The quantity of extracted matter may be reported as mass extracted per mass of wiper or mass extracted per unit area of wiper.

The organic and inorganic non-volatile residue may be further analyzed, when it is desirable to know how much of a particular species is present. Typically, the non-volatile residue is tested for various inorganic, anionic or cationic constituents, for example Al, Ca, Cl, F, Li, Mg, K, Na and Zn (IEST-RP-CC004.2 §6.2).

The invention may be further understood by reference to the following examples.

Example 1: Wipers Coated With Hydroxypropylcellulose

Wipers measuring 9" x 9" and weighing about 4 oz/yd², were knitted from continuous filament polyester yarns (70 denier/34 filament and 70 denier/100 filament yarns in a 3:1 ratio, respectively). The wipers were wet in a 0.1% aqueous solution of hydroxypropylcellulose (average m.w. ~ 370,000), with the excess squeezed out by hand, to achieve an add-on of 0.2 wt.% polymer. The wipers were dried in a forced air oven at 350 degrees F for 6 minutes. Coated wipers were tested for particle attraction to various particles using the particle attraction test described above, and compared to uncoated wipers. The results are shown in the table below.

Particle	Particle Size (μm)	Uncoated Pickup (mg)	Coated Pickup (mg)
Carbon Black	3	13	33
Aluminum Oxide	0.3	8	19
Manganese Oxide	0.4	10	17
Titanium Dioxide	1.3	4	14
Zinc Oxide	2.2	8	25
Aluminum	2.7	8	18
Graphite	3.4	23	32
Iron	1.1	9	26
Ferric Oxide	3.1	6	18
Zinc	1.2	17	22
Ferrite	1.3	8	13
Silicon Dioxide	1.8	12	28

Example 2: Wipers Coated in Washer With Hydroxypropylcellulose

Three-Hundred wipers (9" x 9"), made from the fabric described in Example 1, were wet with 0.6% aqueous solution of hydroxypropylcellulose (average m.w. ~ 110,000), with the excess squeezed out by hand, to achieve an add-on of 1.2 wt.% polymer. The wipers were dried in a conventional laundry dryer for 40 minutes. Some of the wipers were tested for attraction to carbon black particles and found on average to pick up 24 mg. Other wipers were washed in a pilot scale (35 lb. washer) cleanroom laundry using a standard procedure, tested for particle release using the Biaxial Shake Test, and found to have 13 million particles/sq. m. greater than 0.5 microns.

Example 3: Wipers Jet Coated With Hydroxypropylcellulose

Ten wipers (9" x 9"), made from the fabric described in Example 1, were placed in a Werner Mathis CH-8155 laboratory jet in a 0.1% aqueous solution of hydroxypropylcellulose (mol. wt. ~400,000) and jetted at 70 degrees C for 20 minutes, to achieve an add-on of 0.2 wt.% polymer. The wipers were dried in a forced air oven at 350 degrees F for 6 minutes, then tested for particle attraction to carbon black, picking up 30 mg.

Examples 4-13: Wipers Hand Coated With Other Polymers

Wipers, made from the fabric described in Example 1, were wet in an aqueous solution of the polymer (solids noted in table), with the excess squeezed out by hand, to achieve an add-on of about 0.2 wt.% polymer. The wipers were dried in a forced air

oven at 350 degrees F for 6 minutes. Coated wipers were tested for particle attraction to carbon black using the particle attraction test described above, and compared to uncoated wipers. The results are shown in the table below.

Polymer	Carbon Black Pickup (mg)
None (comparative)	10
sodium carboxymethylcellulose (0.1%)	26
poly(carboxylic acid) (0.1%)	27
hydroxypropylcellulose (0.1%)	21
poly(vinyl pyrrolidone) (0.05%)	26
chitosan (0.5%) + melamine formaldehyde resin (0.005%)	27
poly(acrylic acid) (0.1%)	28
hydroxypropylmethylcellulose (0.1%)	28
methylcellulose (0.1%)	29
isopropyl amine/methacrylic acid copolymer (0.1%)	36

Examples 14-15

The following example demonstrates the improvement in absorbency realized when a surfactant residue is left on the wiper.

A fabric as described in Example 1 was pad coated with a 2.4% poly(vinyl alcohol) aqueous solution, vacuumed over a vacuum slot and dried in a tenter frame oven at 250° F to achieve an add-on of 2.1 wt.% polymer. The fabric was laser cut into 9" x 9" wipers and laundered in a conventional cleanroom washer and dryer.

The rate of sorption of the wipers was measured according to IEST-RP-CC004.2 §7.2 (the time required for the disappearance of specular reflection from a drop of water on the surface of the wiper), and found to be 7 seconds.

The wipers were placed back in the cleanroom washer and run through an abbreviated wash cycle in which a commercial detergent, comprised of nonylphenol ethoxylate, was added in the penultimate rinse cycle. The wipers were dried in a cleanroom dryer to achieve an add-on of about 1 ppm surfactant, in addition to the previously applied polymer coating. The rate of sorption was tested as above and found to be 0.2 seconds.

The foregoing examples demonstrate the efficacy of the wiper having a particle attracting finish in absorbing and retaining particulates, especially those in the target range of 0.5 to 20 microns. The wipers attract particulate in both a wet and dry environment, especially in an aqueous environment, such as might be encountered when using a wiper saturated with a solvent. The wipers are not tacky, and the polymer coating does not leave a residue when the wiper is used to clean a surface, wet or dry.

Additionally, by following the teachings of the present invention, it is possible to manufacture wipers which not only have superior cleaning characteristics, but also meet the requirements for Class 1, Class 10, Class 100, Class 1,000, Class 10,000 and Class 100,000 cleanrooms as defined in Federal Standard 209 E. In particular, the wipers meet one or more of the following objectives: to reduce particulate contamination of particles

greater than 0.5 microns to a level of less than 75 million/meters², preferably less than 30 million/meters², as measured by the Biaxial Shake Test (IEST-RP-CC004.2 § 5.2); to reduce particle contamination of particles greater than 5 microns to a level of less than 1 million/m², preferably less than 300,000/m², most preferably less than 150,000/m², as measured by the Biaxial Shake Test (IEST-RP-CC004.2 § 5.2); to reduce non-volatile residues with water extraction to less than .005 grams/meters², and even less than .003 grams/meters² as measured by short term extraction (IEST-RP-CC004.2 §6.1.2); and to achieve absorbance capacities of 3.5 milliliters/meters² or greater, and even 4.0 milliliters/meters² or greater (IEST-RP-CC004.2 §7.1).

A further advantage of the wipers of the present invention is that the particle attracting polymer is believed to reduce particle release from the wiper, such as low molecular weight polyester, which migrates to the surface of polyester fiber. Without being bound to a particular theory, it is believed that the particle attracting polymer may work as a barrier to trap particulate contaminants from being released by the wiper.

The cleanroom wipers find utility in virtually any environment where a low contaminate, high absorbance wiping cloth is desired, such as in semiconductor, optical, food packaging and pharmaceutical cleanrooms, and in preparation of surfaces for painting or other coating.

There are, of course, many alternate embodiments and modifications of the invention, which are intended to be included within the scope of the following claims.

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